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ON EXPLORING THE COMMERCIAL POSSIBILITIES OF
THIN FILM GROWTH AND MATERIALS PURIFICATION
IN SPACE] Annual Report, 1987-1988 (Houston
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SVEC— STATEMENT AND PURPOSE

THE SPACE VACUUM EPITAXY CENTER (SVEC) at the University of Houston is a NASA research center, funded by both NASA and industry, developing techniques to utilize the ultra-vacuum of space for materials processing. SVEC, established by an initial grant of \$5.5 million from NASA, was chosen from among a large number of research universities and institutes to become one of 16 NASA Centers for the Commercial Development of Space.

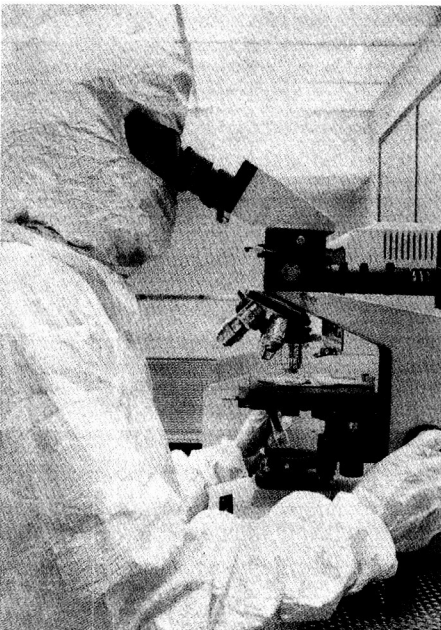
The Center focuses its primary research efforts on exploring the commercial possibilities of thin film growth and materials purification in space. Procedures developed at SVEC, including molecular and chemical beam epitaxy (MBE and CBE), offer visible techniques for producing new electronic, magnetic, and superconducting thin film materials and devices that have properties made possible only by the extraordinary quality of the space ultra-vacuum.

The goals of SVEC include:

- Adaptation of MBE/CBE technology to realize the commercial exploitation of the space ultra-vacuum
- Stimulation of U.S. industrial space research and development
- Assurance of technical support and industrial synergism in the development of space ultra-vacuum thin film technology
- Education and training of scientists and engineers for future space research and manufacturing efforts
- Leadership in the development of new programs in space processing

SVEC serves as a liaison between the private sector and NASA in the nation's endeavor to commercialize space. It will provide commercial opportunities for corporations and exciting research and development opportunities for scientists and engineers seeking to exploit the unique possibilities of the ultra-vacuum of space.

Researcher Susan Street examines a GaAs sample prior to growth.



1987-88
ANNUAL REPORT
SPACE VACUUM EPITAXY CENTER (SVEC)

THE UNIVERSITY OF HOUSTON

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Robert L. Lineberry, Ph.D., Senior Vice-President/Provost

SVEC ADMINISTRATION

Director
Alex Ignatiev, Ph.D., University of Houston

Founding Director
C. W. Paul Chu, Ph.D., University of Houston

Associate Director for Research
Joseph Greene, Ph.D.,
University of Illinois-Urbana

Administrative Director
Alvin F. Hildebrandt, Ph.D.,
University of Houston

Project Administrator
Michael Owens, University of Houston

Administrative Secretary
Alyce Klinger, University of Houston

SVEC
University of Houston
Houston, TX 77204-5507
(713) 749-3701
FAX (713) 747-7724

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SVEC

is on its way to being a self-sufficient research facility in the city of Houston. Space shuttle flights have been restored. Planning is forging ahead on the space laboratory and orbiting platform. We are studying theories and developing the types of equipment that will be needed to synthesize new materials and fabricate novel microdevices in the vacuum of space. The SVEC consortium, already comprised of leaders in the field, expects to admit new members as we realize the commercial use of space.

Little more than a year has passed since the SVEC laboratories were dedicated. The labs now house sophisticated vacuum chambers and equipment for the epitaxial thin film growth of semiconductors, metals, and superconductors.

In order to grow the best possible epitaxial films at the lowest possible temperatures on earth, we are isoelectronically doping material during growth. While we have not yet documented why this works so well, several theories are under study. We have found that isoelectronically-doped film shows the highest mobility in comparison with films grown at optimal temperatures. We have also had success in growing epitaxial films of *InSb* on sapphire which shows promise for infrared sensitive devices in the III-V semiconductor system.

Our laboratory facility now houses an atmospheric scanning tunneling microscope (STM), and development has already begun on two ultra high vacuum (UHV) designs that will provide images of the surface of epitaxially grown thin films by telemetry when we begin manufacturing materials in space. We have proven that we can employ the technique of Pulsed Laser Evaporation to grow superconducting thin films of the new *BiCaSrCuO* material.

Because we expect to grow epitaxial materials in orbiting facilities, we are focusing considerable attention on computer technology. These will be aloft for long periods of time without human supervision. Therefore, we have a paramount need for systems that control the growth process and monitor the quality of materials in space.

Our anticipation of the future makes us feel as though we are already pioneering in space. What we achieve in SVEC laboratories at the University of Houston and in the laboratories of consortium members must ultimately translate to an environment hostile to man.

We were founded in 1987. In 1988, we established objectives and carried out preliminary research. In subsequent years, we will map achievements extending beyond the tracks laid by early investigators. The new pathways we make for ourselves will provide access roads years from now when the commercial world is able to capitalize upon basic research at SVEC.

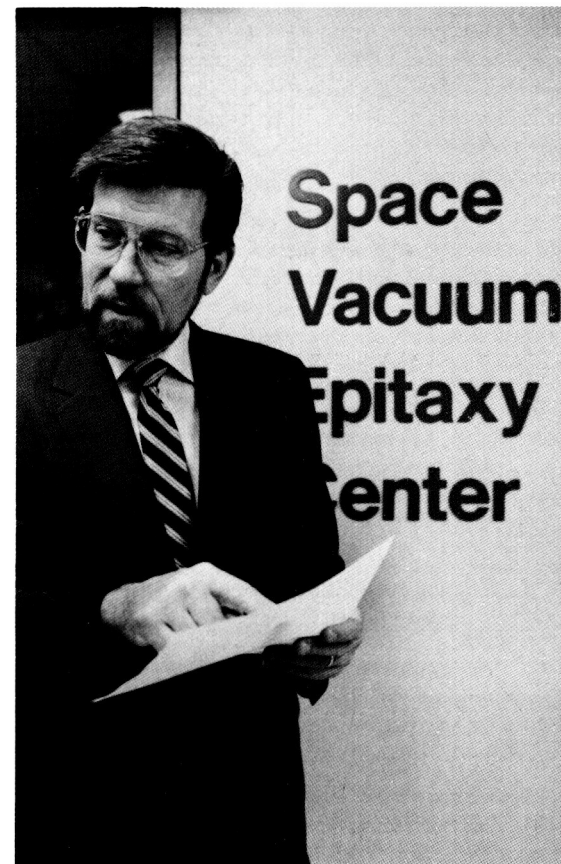
Research reported in this document identifies the challenges researchers have accepted. We are engaged in research aimed at producing thin film semiconductor and superconductor materials in space. All that we are attempting is new. Thus, we not only have to confirm our theoretical position, but we must develop the facilities and the means for getting to space to grow the unique thin film materials.

The 1987-88 SVEC Annual Report may be likened to a map. We know the safe roads to travel. Some are in transition—either in need of repair, under construction, or about to be commissioned for travel. Other routes are in the planning stages. Sparse regions are located on the map and contrast markedly with the street patterns of settled neighborhoods. Through these unpopulated regions, civilization is marked only by untrammelled plains transversed by single railroad tracks that slice through the land.

The SVEC map is a sparse plain. Our excursion into this largely unexplored territory takes us along thin tracks and through new paths in fields of research that characterize ultra high vacuum space research. Others will follow our lead.

—Alex Ignatiev
Director

MESSAGE FROM THE DIRECTOR



*Alex Ignatiev, Director of the Space
Vacuum Epitaxy Center.*

INTRODUCTION TO SVEC SCIENCE AND THE WAKE SHIELD FACILITY

THE SPACE VACUUM EPITAXY CENTER is the center for the development of thin film materials, both natural and artificially structured, in the vacuum of space. These thin film materials are grown by molecular beam epitaxy (MBE) and chemical beam epitaxy (CBE). The term *epitaxy* refers to the growth of crystalline films in which the substrate determines the crystalline composition and orientation of the grown layer. Molecular beam *epitaxy* is the growth of films through the use of one or more thermal molecular beams for the deposition process, while chemical beam epitaxy is thin film growth through the chemical reaction of gaseous chemical beams with a crystalline substrate.

The term heteroepitaxy refers to the growth of crystalline films on dissimilar substrates. With heterostructures, we have films consisting of dissimilar layers grown on top of each other. A superlattice is a layering of periodic heterostructures. These epitaxial layers are grown under ultra high vacuum conditions. In the controlled epitaxial deposition of atoms onto a substrate, atoms are literally laid down in an atom-by-atom, layer-by-layer manner to form the required films. On earth, vacuum chambers with their inherent limitations in ultimate vacuum, size, and pumping speed are used to grow films. Space, however, offers vacuum possibilities not available terrestrially. By harnessing the vacuum of space to help us develop epitaxial thin films, we can function in a laboratory of infinite proportions where research and development opportunities are limited only by our imaginations.

MBE/CBE technology is generally considered to be the most powerful technique for the synthesis of new materials and the fabrication of novel micro-devices. It has applications in opto-electronics (lasers and detectors), in microwave amplifiers (low-noise and high-power), in millimeterwave sources and amplifiers (30 GHz to 300 GHz), in high speed digital logic and memory

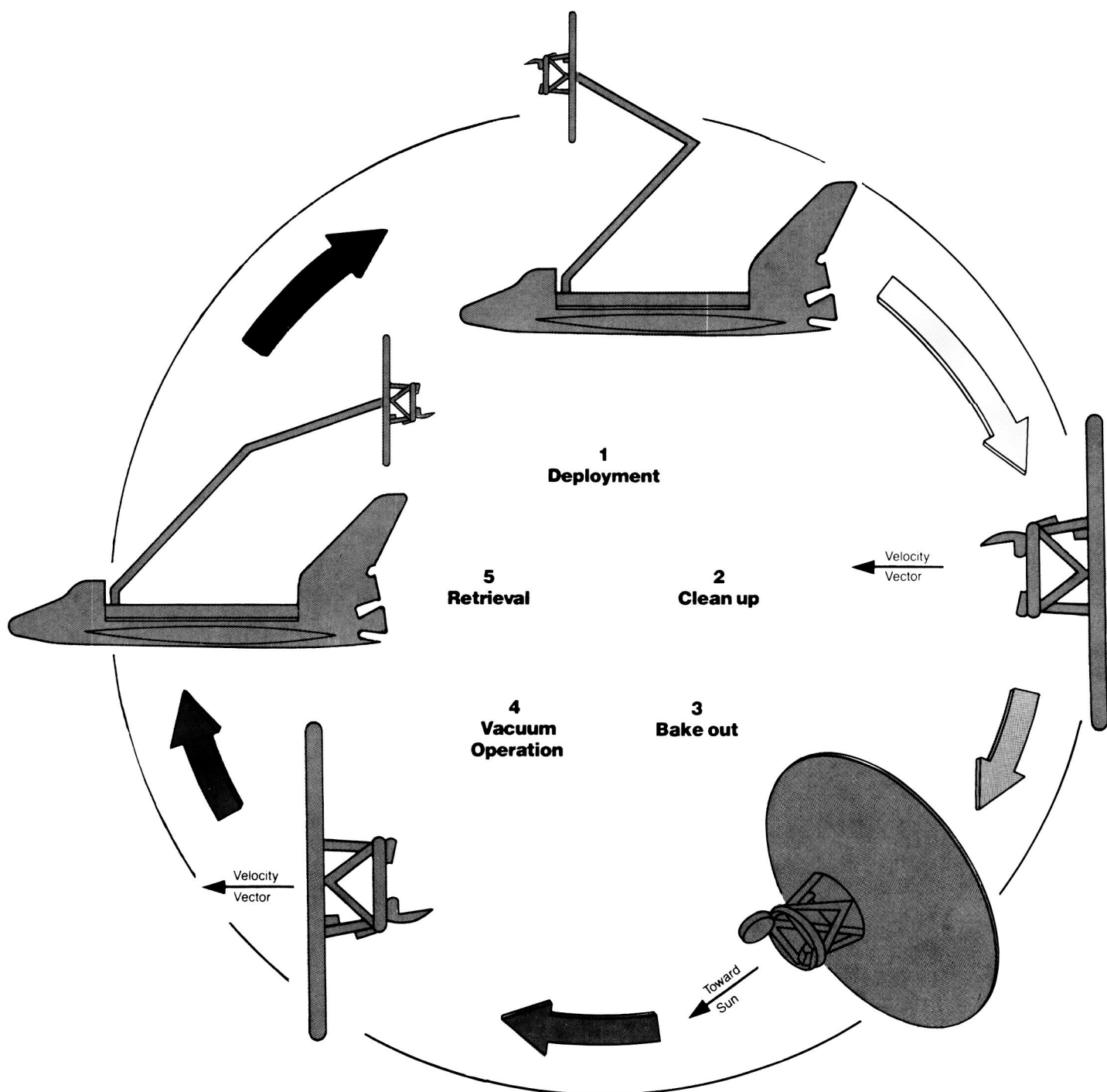
elements, and in thin film, high current density, high-temperature superconductors. SVEC is utilizing the terrestrially-based MBE/CBE technology in a number of these application areas described in the following pages. The major focus of SVEC, however, is the space-based epitaxial growth of thin films. To this end, SVEC has embarked on an ambitious program of flight hardware design, construction, and deployment. The flight hardware is a circular shield, 12 feet in diameter, defined as the *Wake Shield Facility* (WSF), which will be flown on the shuttle and deployed on the shuttle arm. Deploying this wake shield, SVEC scientists expect to produce epitaxial semiconductor and superconductor thin films of higher quality than those produced on earth.

Development of a Wake Shield Facility, formerly SURF (Space UltraVacuum Research Facility), is continuing at SVEC—University of Houston, beginning with a proposal to NASA to accomplish an initial flight demonstration of thin film epitaxial growth in space on the shuttle. This proof-of-concept experiment will be an MBE growth of compound semiconductor thin films, which will define the quality of achievable products.

The current design for the WSF is a commercial low-cost approach to the development of flight hardware through a series of contracts to industry, including consortium members, and will assure cost-effective, reliable hardware. Growth experiments will enable limited characterization of the wake shield ultra-vacuum environment. The overall concept for the WSF flight program involves the use of the STS Remote Manipulator System (RMS) to position the WSF in a favorable location relative to the orbiter. The complete configuration includes the MBE process system, the structural and mechanical system, an instrumentation and signal distribution system, a data acquisition and control system, and an electrical power system. The first flight of the WSF is expected in the fall of 1991.

WSF—Wake Shield Facility Operation. With the wake shield, scientists can produce thin film materials by utilizing the ultra vacuum of space as a high quality work environment. On earth, such innovative production of materials is limited by the constraints of Clean Room facilities and restricted by the steel walls of a vacuum chamber. Deployed by the Space Transport System (the space shuttle) in a low earth orbit, the shield pushes aside stray atoms and molecules to create an ultra vacuum in its wake. By this technique, the whole of space becomes a massive laboratory.

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SVEC CONSORTIUM & MEMBERSHIP

THE SPACE VACUUM EPITAXY CENTER is a consortium located at the University of Houston in Houston, Texas. It is supported by a NASA grant to the university with accompanying research support from university, industrial, and governmental agencies. The consortium is dedicated to the commercialization of space. Its primary function is the utilization of space ultra-vacuum for both thin film processing and the purification of materials. SVEC invites industrial and commercial interests to join the consortium as full members or as associate members.

Current members of the consortium are from industry, government, and academia:

AT&T Bell Laboratories
Electro-Optek, Inc.
Instruments S.A., Inc.
Perkin-Elmer
Rockwell International
U.S. Army Laboratory-Watertown
University of Houston
University of Illinois-Urbana

Full membership entitles a participant to four basic benefits:

- Opportunity for the development of SVEC patents
- Access to SVEC technical information
- Access to SVEC facilities and personnel
- Representation on the Center Development Committee

Associate membership entitles the member to the following benefits:

- Access to SVEC publications prior to general dissemination
- Receipt of SVEC semi-annual and annual reports
- Invitations to attend SVEC symposia, lectures, and other functions
- Personal interaction with full members

Consortium members have a financial obligation which they regard as an investment in future productivity. Full members must agree to a yearly contribution equal to \$50,000, with a minimum cash outlay of \$15,000 and the remainder in direct in-kind support. Associate members are required to make an annual cash contribution of \$5,000.

Administrative Structure

SVEC is managed by a Center Directorate and a Center Development Committee.

Center Directorate:

The Center Directorate is responsible for administrative operations and the conduct of research and development in the center. Authority rests with the director, appointed by the Senior Vice President/Provost of the University of Houston.

Director: Paul C. W. Chu, University of Houston (through April, 1988)

Director: Alex Ignatiev, University of Houston (from May 1, 1988 to present)

Associate Director for Research: Joseph Greene, University of Illinois-Urbana

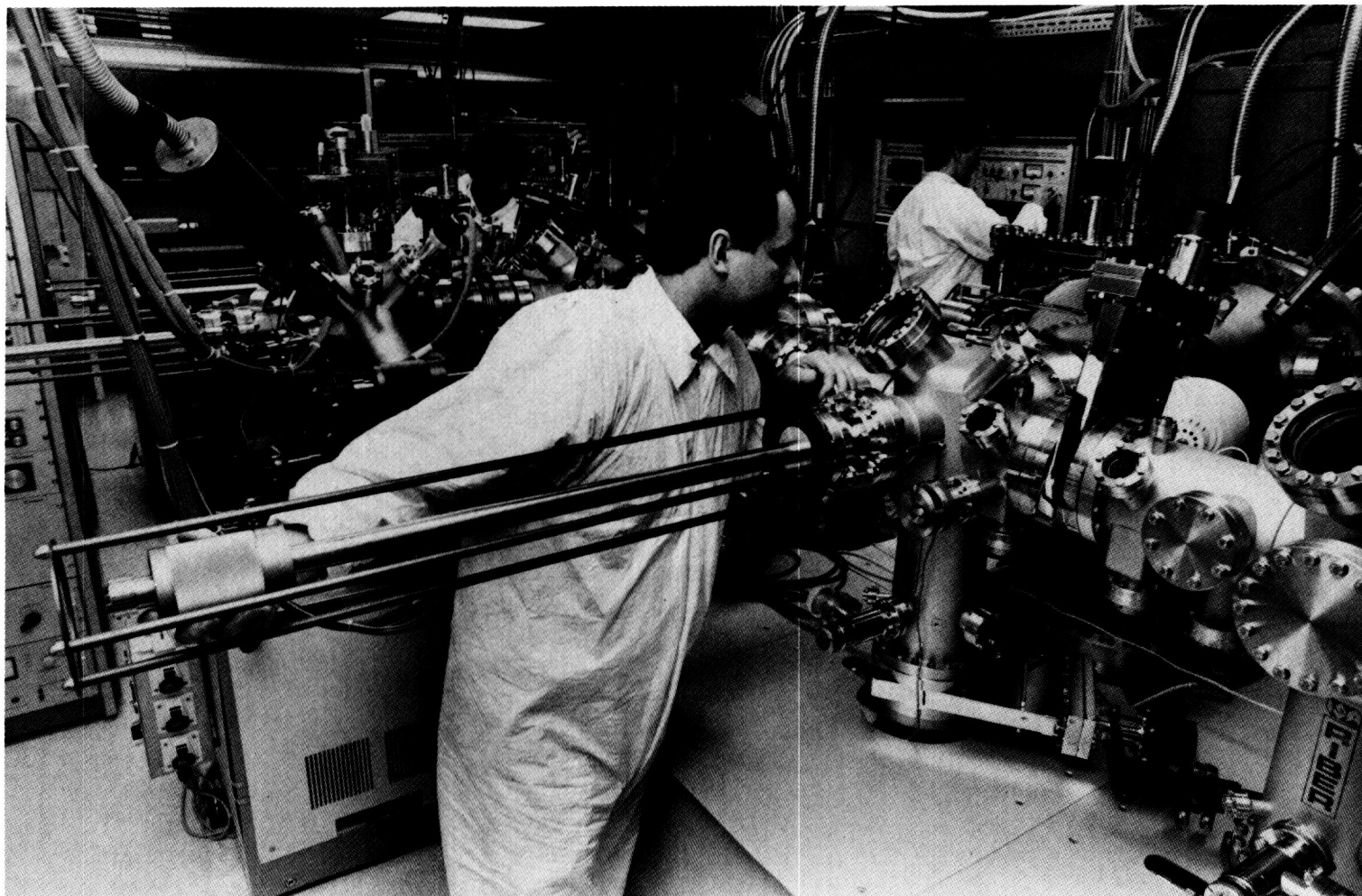
Administrative Director: Alvin F. Hildebrandt, University of Houston (through August 31, 1988)

Project Administrator: Michael Owens, University of Houston

The Directorate is supported by staff including:

Laboratory Manager:	Robert Keith
Administrative Secretary:	Alyce Klinger
Accounting Specialist:	Larry DeBondt

The Directorate of the center has access to the vast expertise at NASA-MSFC and NASA-JSC, as well as the talents of the SVEC industrial members and the academic communities at the University of Houston and the University of Illinois.



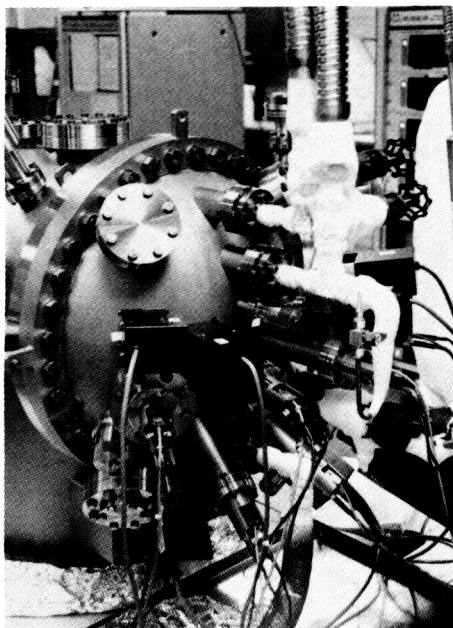
Researcher Keith Jamison transfers a GaAs sample into the Chemical Beam Epitaxy (CBE) chamber.

Center Development Committee:

The Center Development Committee, comprised of representatives of the member organizations in conjunction with the Directorate, will define overall policy and direction of the center to ensure that goals and objectives remain industry driven. The committee will work towards establishing membership criteria intended to convert the center into a self perpetuating organization. The Center Development Committee functions with four subcommittees:

- Oversight Subcommittee — monitors the execution of research projects
- Patent & Royalty Subcommittee — deals with trade secret policy and resolves disputes arising from patent credits or patent assignments
- Policy Subcommittee — establishes center operation policy and membership guidelines
- Strategic Planning & Marketing Subcommittee — determines the optimum use of space ultra-vacuum benefits and the use of SVEC and NASA facilities

MOLECULAR BEAM EPITAXY



MBE Evaporation Chamber - The chamber shown above has been prepared for the epitaxial growth of III-V compounds.

RESEARCHERS AT SVEC are investigating the growth of III-V semiconductors in space by first studying the growth of high quality epitaxial films at the lowest possible temperature in our earth-based MBE system. Low temperature growth enables researchers to make abrupt interfaces and eliminate interdiffusion in heterostructures. The highest quality MBE *GaAs* epilayers are grown at $\sim 600^\circ\text{C}$. Growth of epilayers at lower temperatures, although possible, yields material with poor electrical properties.

MBE-Isoelectronic doping of GaAs and GaAlAs

One successful method of achieving lower temperature growth without sacrificing electrical quality is the isoelectronic doping of the material during growth. For example, indium doping during *GaAs* bulk growth yields lower defect wafers. Preliminary studies have shown that both indium and antimony may be useful as an isoelectronic dopant in MBE growth of *GaAs*.

During the last six months, SVEC personnel have studied and characterized the influence of small amounts of the isoelectronic dopant antimony on MBE growth conditions, morphology, and electronic properties (such as deep level traps, photoluminescence, and Hall mobility) of *GaAs(100)* and *AlGaAs(100)* thin films. These studies have had two purposes: (1) growth of a high quality material at lower than normal growth temperatures with isoelectronic doping and (2) understanding the physics behind isoelectronic doping.

A major diagnostic tool used in this study is deep level transient spectroscopy (DLTS) for characterizing the deep level traps in semiconductor materials. Researchers at SVEC established a fully tested and operational DLTS facility. Metalization equipment for making contacts to the semiconductor materials has also been added to the facility.

In the current study, *n* doped *GaAs(100)* and *AlGaAs(100)* were grown at various temperatures and under various isoelectronic dopant conditions. The electrical properties and morphology of the different growths were compared to material grown under normal growth conditions. Preliminary results of this study, presented at the 35th AVS meeting in Atlanta, Georgia, indicated that isoelectronic antimony doping reduces deep level traps for materials grown at 50°C below their normal growth temperature. This may have significance in the growth of *GaAs/AlGaAs* superstructures by allowing growth to occur at a constant temperature without compromising the electrical properties of either material.

MBE-Study of defects in semi-insulating GaAs(100)

SVEC consortium members at the U.S. Army Materials Technology Laboratory have been studying single-crystal semi-insulating *GaAs* materials. The quality of semi-insulating *GaAs* is directly related to the concentration of EL2 defects. The EL2 trap is believed to be a mid-gap double donor defect complex which is based on an arsenic atom occupying a gallium vacancy (antisite defect) in a complex with an excess interstitial arsenic atom. If this trap is present in semi-insulating *GaAs* to be used as a substrate at concentrations exceeding 10^{16}cm^{-3} , devices grown on this substrate will suffer intolerably high I/F noise.

Certain characteristics of the EL2 defects have been determined, including two trap-liberation time-constants, the transition of EL2 to its low-temperature metastable state (EL2*) in the temperature range 110-130K, and the persistent photoconductivity identified with EL2 compensation single crystal *GaAs*.

This study, in conjunction with other work, has determined that the concentration of EL2 can be precisely controlled by proper growth techniques, including inverted thermal conversion.

MBE-Growth of InSb(111) on Sapphire(0001)

From the perspective of pure science and for the development of remote sensing tech-

nology, researchers have shown interest in photodetectors that can detect light in the 3-5 micron and 8-12 micron wavelength regions. At these wavelengths, light can travel with little adsorption through the atmosphere. Most of the work to develop these photodetectors has focused on II-VI semiconductors comprised of $Hg_xCd_{1-x}Te$, where x is adjusted to the appropriate wavelength. However, the stability of $HgCdTe$ is inferior to semiconductors made from III-V materials. Recent theoretical predictions based on a strained layer superlattice of the III-V material $In_xAs_{1-x}Sb$ have predicted that this material may be a good substitute for $HgCdTe$ in both the 3-5 micron and, possibly, the 8-12 micron wavelength range.

Growth of a ternary strained layer superlattice usually starts with the binary III-V material, adding the third material during growth until the proper ratio of the compounds is attained. However, one of the problems with using $InSb$ or $InAs$ as a starting material for the growth of $InAs_xSb_{1-x}$ is that neither $InSb$ nor $InAs$ is currently produced in wafer form in larger than a one inch diameter, and neither has good mechanical properties. Sapphire, on the other hand, has both good thermal and mechanical properties; it is transparent up to 5 microns; and the (0001) face has a four percent lattice mismatch to the $InSb(111)$ surface. $CdTe(111)$ has been successfully grown on sapphire and $InSb$ is very similar in atomic spacing to $CdTe$. These facts and success with the growth of $CdTe(111)$ on sapphire by one of our consortium members motivated us to look at the growth of $InSb$ on sapphire as the starting point for later growth of $InAsSb$ strained layer superlattices.

Researchers at SVEC in conjunction with scientists at Electro-Optek Corporation have, for the first time, successfully grown epitaxial layers of $InSb(111)$ on sapphire (0001). The quality of the epilayers is dependent on the flux ratios and temperature of the sapphire substrate. As a result, researchers are attempting to optimize the growth conditions. Scientists at the University of Illinois have analyzed a number of the samples using transmission electron microscopy (TEM). TEM micrographs of the $InSb/sapphire$ interface show good epitaxy with some microtwins and small antimony precipitates at the interface. Quality should improve with

further experience in growth of this structure.

Mobility and DLTS measurements of this surface are in progress.

MBE-Silicon epitaxy

Perkin-Elmer, a SVEC consortium member, has established a new silicon MBE laboratory. This facility allows SVEC researchers to fabricate and study thin epilayer structures of silicon and related compounds.

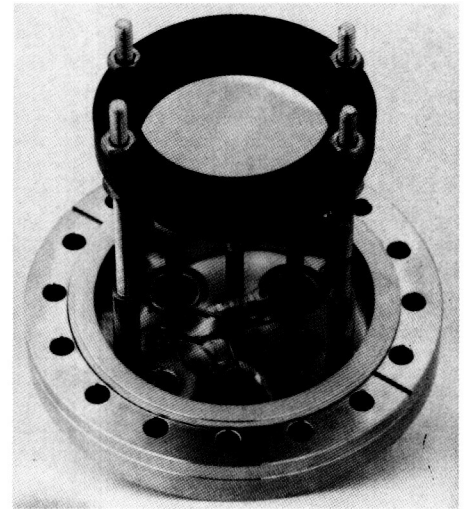
Strained layer Si/Ge superlattices and multiple (Si_mGe_n) structures have been grown and have been shown to have high structural perfection and mono-atomic layer control. The doping control in these materials is being investigated.

The work at Perkin-Elmer has established a benchmark for this class of new materials. Heterojunction Bipolar Transistors (HJBT) and other advanced structures of Si/Ge are now possible. These novel structures offer promising device performance that will have wide applications in the electronics industry.

MBE Novel Source Design

We have completed the design and proof-of-concept phases for a surface-tension-containment evaporation source. This source consists of a disk of material, electron beam-heated from the back surface, in which evaporation occurs from the front face of the disk. The design has been shown to produce a 0.5 cm diameter melt in Si at a temperature in excess of $1400^\circ C$ significant evaporation occurring. The deposition rate was found to be approximately .5 microns per hour at a distance of 15 cm from the melt. Heating larger areas produced perforation of the melt in the first attempt. The perforation may have been related to gravitational effects or to vibrations. In either case, such problems can be expected to be significantly reduced in the WSF environment. Further characterization experiments aimed at design optimization are currently underway, and a patent application is being drafted. Publication of the results is anticipated in 1989.

Other evaporation source design efforts have concerned characterization of the properties of an ion-beam source and the changes in properties of dopants in Si produced by ion-assisted techniques compared with the properties of thermally-evaporated dopants.



Surface-Tension Containment Evaporator.

CHEMICAL BEAM EPITAXY GROWTH OF III-V COMPOUNDS

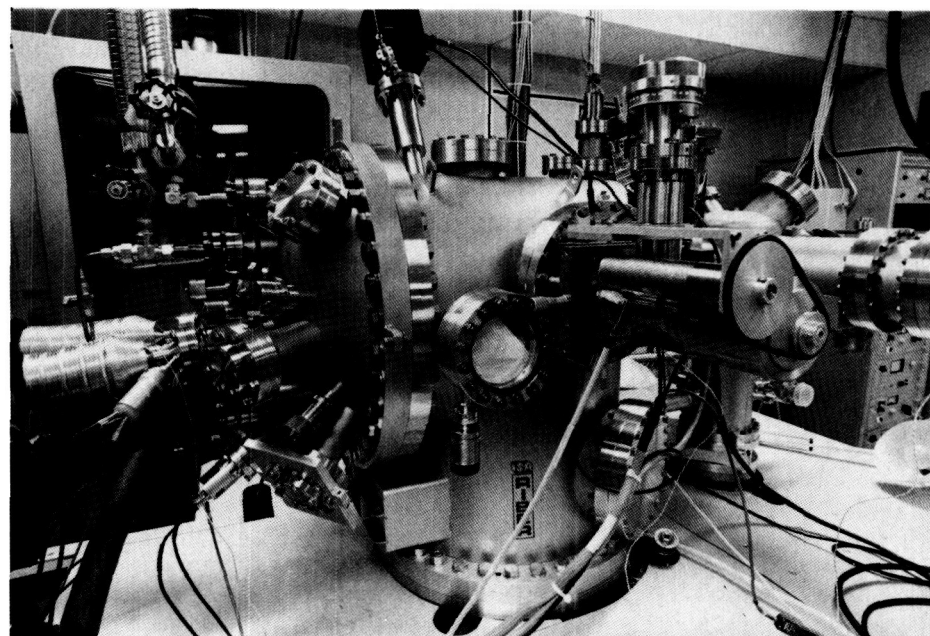
THE CHEMICAL BEAM EPITAXY SYSTEM (CBE) installed at SVEC in early 1988 as one of the first commercially available CBE systems in the U.S. is now operational. Triethylindium ($TEIn$), triethylgallium ($TEGa$), and Arsine (AsH_3) are available for the growth of epilayers and have been used to date to grow $GaAs$ and $InGaAs$. Grown films have the appearance of a mirror finish with oval defect counts below $10/cm^2$. Hall effect and photoluminescence measurements on these samples are not yet available.

To permit the further growth of a wider range of materials and structures in the CBE system, we are adding a third OM line for an aluminum source ($TMAI$) and a second hydride line for phosphine. With these modifications, the additional growth of GaP , InP , and $AlGaAs$ epilayers will become possible.

In collaboration with ISA, Inc., researchers are in the process of modifying the existing gas inlet system to improve the growth rate control and to minimize the dead volume in gas cells in the present configuration.

In current efforts, researchers have measured the arsine cracking efficiency in the "high temperature gas cell" at different temperatures by a mass spectrometer mounted on the growth chamber. Both hydrogen and arsine peaks were monitored by a mass spectrometer mounted on the growth chamber as a function of the cracker cell temperature.

Researchers completed a series of $GaAs$ growth experiments with varying $TEGa$ flow rates at a constant AsH_3 flow rate. The growth rate was determined by a computer-



A side view of the Modutrac, a component of the epitaxy system for the growth of thin films.

ized video RHEED system which measured RHEED oscillations. The growth rate is roughly linear with $TEGa$ flow in the range of 2 sccm to 15 sccm. The RHEED oscillations period, measured as a function of growth time, varied by as much as 100%, as calculated from the period of the first RHEED oscillation and the final equilibrium value. This observation suggests that the growth of superlattice structures in the present configuration will still require the use of mechanical shutters for precise growth control; otherwise, corrections for the growth rate changes must be implemented through the computer control software.

Researchers have also investigated the crossover from As to gallium-rich growth as a function of AsH_3 and $TEGa$ flow. They found that for an AsH_3 flow as low as 2.5 sccm (5% of max), the crossover to Ga-rich growth does not occur until a 4 sccm flow (70% of max) of $TEGa$ is attained. Results suggest an abundance of As species at the substrate surface.

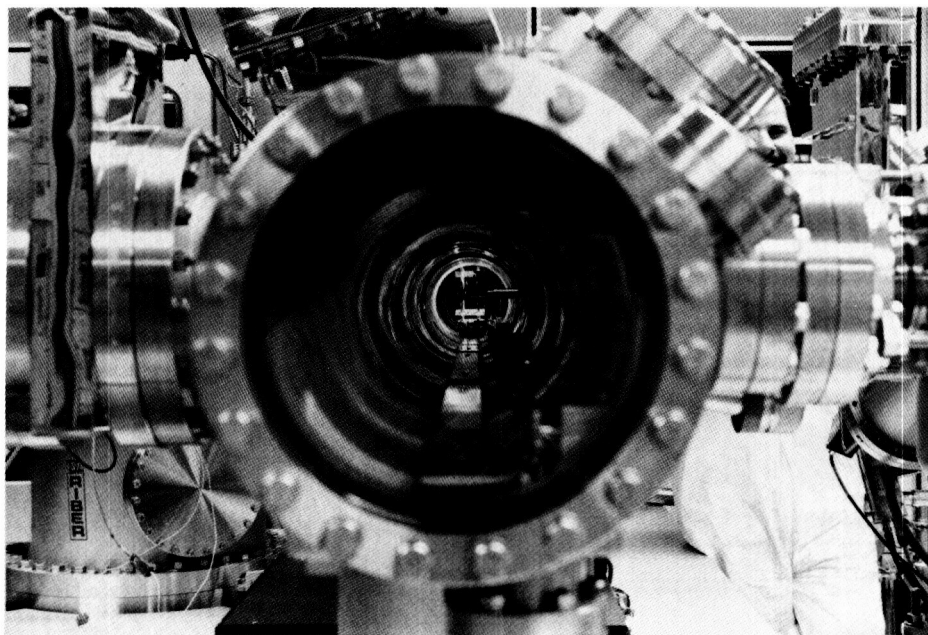
Further work is underway to optimize growth conditions. Researchers need to investigate parameters such as bubbler bath temperature, "low temperature" cell temperature, and H_2 flow rates through the bubblers.

REFLECTION high-energy electron diffraction (RHEED), a technique like the more familiar method of low-energy electron diffraction (LEED), is used to determine the structure of crystalline surfaces. The forward scattering geometry of RHEED makes it ideal for combining with molecular beam epitaxy (MBE) of semiconductors, where it has become the standard *in situ* probe for epitaxial growth.

The most widely used aspect of RHEED during MBE is the phenomenon of oscillations in the intensity of the specular beam. It has been well established that the period of these oscillations corresponds exactly to the growth of one monolayer when epitaxy proceeds in a nucleation and growth mode. RHEED oscillations during MBE are thus routinely used to determine growth rate and to calibrate beam fluxes.

Another application of RHEED utilizes "rocking curves." The intensity of the diffracted beams in the RHEED diffraction pattern changes as the incident angle is

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A view inside the Modutrac, a component of the RIBER epitaxy system.

ATOMIC STRUCTURE OF EPITAXIALLY- GROWN FILMS

changed. Through comparison of the measured intensity vs. incident angle rocking curves and theoretical rocking curves, one can determine the exact atomic surface structure of a surface with accuracy better than 0.05 \AA .

Video RHEED System

With growth rate and beam flux calibration as the immediate goal, SVEC researchers have developed a video RHEED system to record RHEED oscillations automatically. The system utilizes a video camera, an IBM compatible personal computer, and custom software to digitize and store the intensities of several diffracted beams simultaneously. SVEC is currently using the technique of RHEED oscillations routinely to calibrate growth rates and has determined flux ratios for AlGaAs and InGaAs heterostructures. In addition, RHEED video data are used in the rocking curve form for atomic structure determination.

Software is being developed to analyze RHEED oscillation data by Fourier methods and to calculate a growth rate automatically. The goal is to feed growth rate information in real time to an expert MBE process control system for the optimization of epitaxial films.

Experimental Investigations

Most practitioners of the RHEED oscillation technique focus only on the intensity of the specular beam. There is a wealth of information, however, that can be obtained from monitoring oscillations in the whole diffraction pattern. We have recorded oscillations in 01, 02, 1/2-order, and 1/4-order beams during MBE growth on the $\text{GaAs}(001)$ surface at various incident and azimuthal angles. Phase differences have been observed between the oscillations of different beams and different decay characteristics of the oscillations. The goal is to obtain quantitative information regarding surface quality and roughness, surface reconstruction, mean island size, and step edge density and orientation.

SVEC rocking curve measurements have focused on the $\text{Si}(111)$ surface, an important semiconductor surface of complex (7×7) reconstruction, and its interaction with metal overlayers. The goal is to determine the nature of the overlayer structure and to study how the metal overlayers affect the substrate structure, as a function of experimental conditions such as temperature and concentration. The first overlayers to be studied are silver and gold. These are

important in applications involving computer I-C chips; their structure is still controversial in the literature.

Theoretical Investigations

SVEC has developed a Monte Carlo simulation of crystal growth that calculates, using the kinematic approximation, the specular beam RHEED intensity at each step of the crystal growth. The model uses a simple cubic lattice and Arrhenius-type surface diffusion of adatoms. We have been able to reproduce general features of crystal growth, such as three-dimensional growth at low temperatures, step-edge propagation at high temperatures, and/or high step-edge densities, as well as decaying RHEED oscillations for epitaxial growth at intermediate temperatures.

We are currently incorporating features specific to GaAs epitaxial growth into the model. These include changing to a diamond "zinc-blend" lattice and modeling the 2×4 surface reconstruction. We will attempt to reproduce particular features of $\text{GaAs}(001)$ epitaxial growth such as phase differences between oscillations in several diffracted beams and decay envelopes. The goal is to better understand the MBE growth mechanism of GaAs and other epitaxial films.

THIN FILM SUPER- CONDUCTORS

THE THIN FILM SUPERCONDUCTOR PROJECT at SVEC is an ambitious undertaking to develop high temperature superconductors for the electronics industry. Our goal is to epitaxially deposit the new generation of oxide superconductors on silicon and gallium arsenide substrates with low temperature processing.

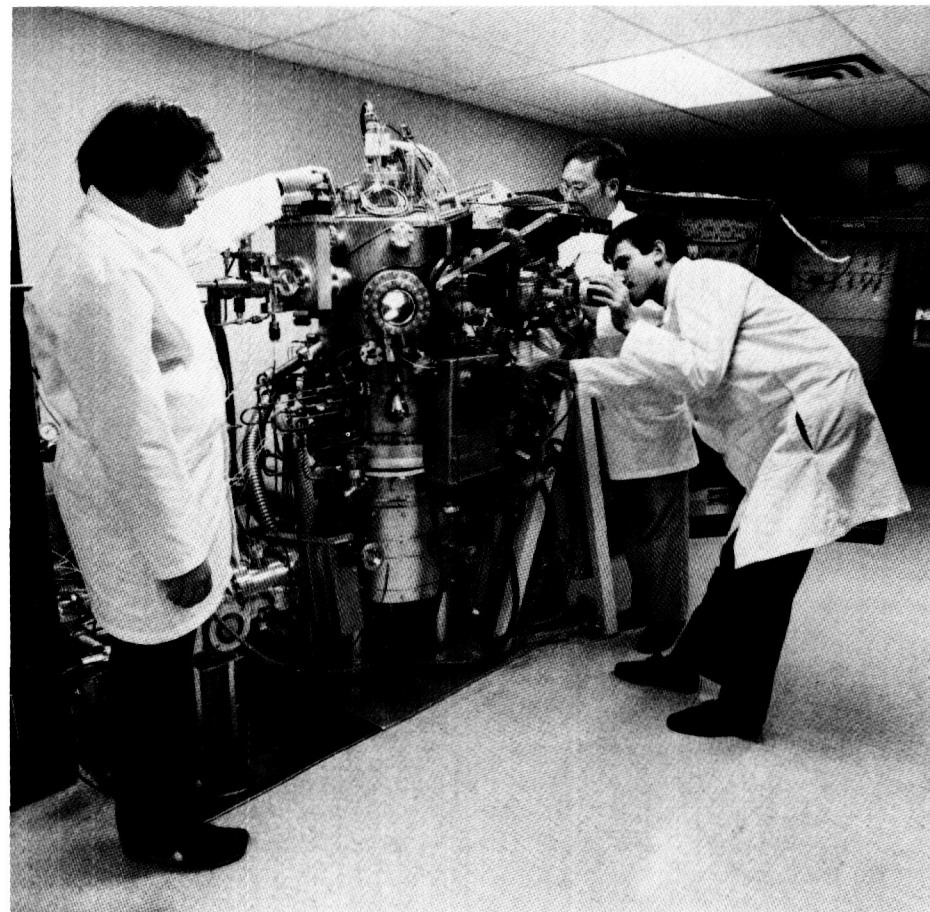
An ultra high vacuum chamber has been constructed for depositing thin films of superconductor material by molecular beam epitaxy. The chamber incorporates a sample transfer system to ensure vacuum quality and *in situ* analysis of films by Auger Electron Spectroscopy and RHEED.

The films are deposited from four evaporation sources: three electron beam evaporators and one resistively heated crucible. Each of the evaporation sources is monitored by a quartz crystal oscillator to control the evaporation rate. The stoichiometry of deposited films is controlled by adjusting the relative rate of evaporation of the four sources.

The greatest difficulty in depositing thin film superconductors lies in achieving correct oxygen stoichiometry. To address this problem, we have developed a special oxygen source. A catalytic process is used to dissociate molecular oxygen to atomic oxygen. The atomic oxygen derived from this technique provides enhanced oxygen incorporation in the films.

Thin film superconductors have also been deposited by laser evaporation of the bulk materials. SVEC researchers have studied the deposition of bismuth superconductors. Researchers at the Rockwell International Science Center have studied the yttrium superconductors. Laser evaporation is a promising technique for depositing these complicated materials.

Laser evaporation is accomplished by focusing a pulsed laser beam onto a pellet of prepared superconducting material. Optical



J. Hughes (l.), J. Liu (c.) and A. Bensaoula (r.) at work on the thin film superconductor growth chamber.

energy is absorbed near the surface of the pellet. The region of absorption rapidly heats and creates a plasma just below the surface. The plasma results in an ablation of the surface material, which, in turn, causes a plume of material to be ejected normally from the surface of the pellet.

The material ejected from the pellet has the same elemental composition as the original compound. Thus, laser evaporation provides a simple means of stoichiometry control. The substrate is placed so that the plume will direct evaporated material toward it. In addition to the evaporation of the metals, it is generally necessary to provide a background of oxygen in the deposition chamber to ensure proper stoichiometry.

Deposition facilities have been constructed at SVEC and at Rockwell. In both systems, the substrate temperature, oxygen pressure, and laser pulse rate can be controlled. The SVEC system uses a point-focused laser beam, while the Rockwell facility uses a raster system to project the laser beam across a rectangular pattern on the target. Both

systems use a flow of oxygen onto the target during laser exposure. Both systems have been characterized and used to produce thin films (1 to 2 microns) of superconducting material. It is always necessary, however, to anneal the films in an oxygen atmosphere at high temperature (850° to 900°C) after deposition to obtain superconductivity.

An investigation has been carried out to study the nature of superconductivity in the 1-2-3 perovskite system. A wide variety of oxide and carbonate powders were used to produce a family of $A\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ compounds. The reaction processes were studied by the techniques of differential thermal analysis (DTA) and thermogravimetry (TG). Researchers found that most of the lanthanides produce similar results.

The following elements were tried: *La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, and Y*. The DTA technique reveals that two non-reversible endothermal processes typically take place during the reaction. The first process takes place at a temperature ranging from 820° to 860°C, while the

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COMPUTER CONTROL OF EPITAXIAL GROWTH

Integrated computer control of the epitaxy process, often an afterthought in previous research, is a fundamental requirement of SVEC's efforts. The orbiting WSF which will carry out production of epitaxial materials in space must be self-governing for long periods of time with little or no human intervention. This space platform will incorporate built-in, rather than added-on, expert control systems that will use the knowledge of human researchers to govern the growth processes. Developing such control systems, using SVEC laboratory and MBE/CBE machines, will be the purpose of the groundbased scientific research. The same equipment will be utilized for control design and testing. Implementation of integrated control methods at SVEC will also yield techniques for ground-up construction of expert control for other space-borne manufacturing efforts.

The computer control program at SVEC is being carried out with an eye for the requirements of space operations. Design of the WSF calls for a self-contained process-control unit (computer and data acquisition/control). The wake shield will receive minimal attention from shuttle crew members other than positioning with the RMS arm.

Once the facility is positioned, the control system will be responsible for all scientific and operational aspects of the mission, including experiment scheduling, adjustment of the growth process, and fault compensation. Initial runs will require about 24 hours of autonomous control, but free-flyer units will eventually operate untended for months at a time, underscoring the need for a versatile and robust control system.

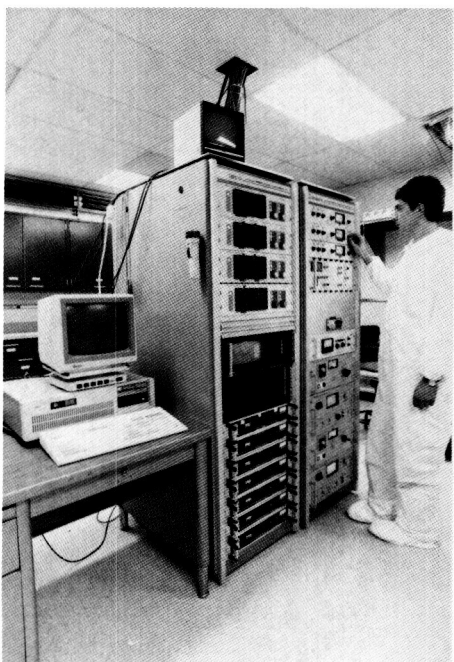
To fulfill this need, expert-system methods will be incorporated into the design of the control system software. An expert system is a program which draws upon the knowledge of a domain expert, (i.e., an experienced specialist in a particular field) and uses this knowledge to make decisions based on qualitative and incomplete data. The knowledge is coded in such a way that the computer "understands" objects and their relationships to one another, and can apply the same rules to a situation that the domain experts use. In this situation, domain experts would include epitaxy researchers, space hardware designers, and space physicists familiar with the low-orbit environment.

These experts will provide the information necessary to prepare software which can handle both the epitaxy process, itself, and possible problems associated with operations in near-Earth space. For example, the control program will use data extracted from electron diffraction patterns observed during growth, combined with knowledge of the growth characteristics of the current material, to adjust deposition rates—a task unsuited for conventional programs. At the same time, the computer will also be monitoring the status of the various wakeshield systems in order to compensate for any problems that arise. For instance, if power were lost to a certain source, the expert controller would decide what goals could still be met for the rest of the mission. The controller could then schedule the growth of alternate materials that do not require use of the failed source.

Development of the control system at SVEC has been underway for approximately a year and has focused on a conventional-system prototype for the Ribier MBE/CBE units.

Currently, a testbed version of the control program is operational for the MBE system. Implementation on the CBE chamber and integration of data from sources such as ion gauges and IR temperature meters are currently being developed.

Autonomy of the WSF is the direct goal of the control system effort at SVEC, but not the only objective. Development of the software should make a measurable contribution to the design of controls for other epitaxy facilities on Earth and also aid in the design of expert control systems for untended space-manufacturing facilities in general.



The gas control panel of the Chemical Beam Epitaxy (CBE) system.

A SVEC technician adjusting MBE system controls prior to switching the system over to computer control.

second process occurs at a temperature that ranges from 890° to 980°C. In addition, some compounds exhibit a reversible endothermal process at a higher temperature.

With the exception of *Ce*, *Tb* and *Pr*, all of the elements investigated will form a superconducting compound with a transition near 90K. Cerium, however, does not form a 1-2-3 compound. This is presumed to be created by the large ionic radius of Ce^{3+} . Praseodymium will form a 1-2-3 compound only with a special gas-exchange technique. The *Pr* compound, however, is not superconducting. Additional work on these compounds is expected to result in a better understanding of the nature of superconductivity in the 1-2-3 crystal.

SCANNING TUNNELING MICROSCOPY

Predicted by quantum mechanics and demonstrated by Meissner and others in the early 1930's, the tunneling of electrons through a potential barrier had long been a fascinating, if not overly useful, physical phenomenon. In 1981, that field of study changed dramatically when Gerd Binnig and Heinrich Rohrer began developing their scanning tunneling electron microscopy (STM) for IBM. Less than eight years ago, Binnig and Rohrer's large and ungainly experimental apparatus gave the first faint hints of individually imaged atoms. Today, the latest generations of the microscope, small and relatively reliable, yield strikingly detailed pictures of the electronic topology of a seemingly endless number of materials in a wide variety of environments.

SVEC, in collaboration with Professor Joseph Lyding and his electrical engineering research group at the University of Illinois, is developing several state-of-the-art scanning tunneling microscopes. While testing and experimentation continue with the University of Houston's first atmospheric STM, construction has begun on two ultra high vacuum (UHV) designs that will eventually provide real-time imaging of the surface kinematics of thin films.

SVEC's STM project draws on a wide range of technology. The microscope scanning and data acquisition are controlled by PC AT computer technology. Extremely high speed D/A and A/D conversion is required. Tunneling current amplification, feedback control, bias circuitry, and sample transport are performed by custom built electronics. The physical microscope requires precision machining of piezoelectric crystals and novel microelectronic assembly techniques. Perhaps the most crucial element of the system is the scanning probe. Microscope resolution is on the order of probe tip sharpness. Designing a reliable procedure

for producing tips of atomic sharpness while under the constraints of UHV, more than any other aspect of the project, puts our efforts on the frontier of technology and theory.

In general, increased efficiency and improvement of the resolution of scanning tunneling microscopy has paralleled reduction in the size of the scanning elements of the microscopy. While the interference of high frequency noise is a serious electronic consideration, it is the low frequency noise near the lower eigenfrequencies of the piezo assembly that can totally obscure information about the surface. In particular, common building vibrations in the 10-100Hz range were a vexing problem for early STM's, resulting in large and complicated vibration isolation systems, some of which filled small rooms.

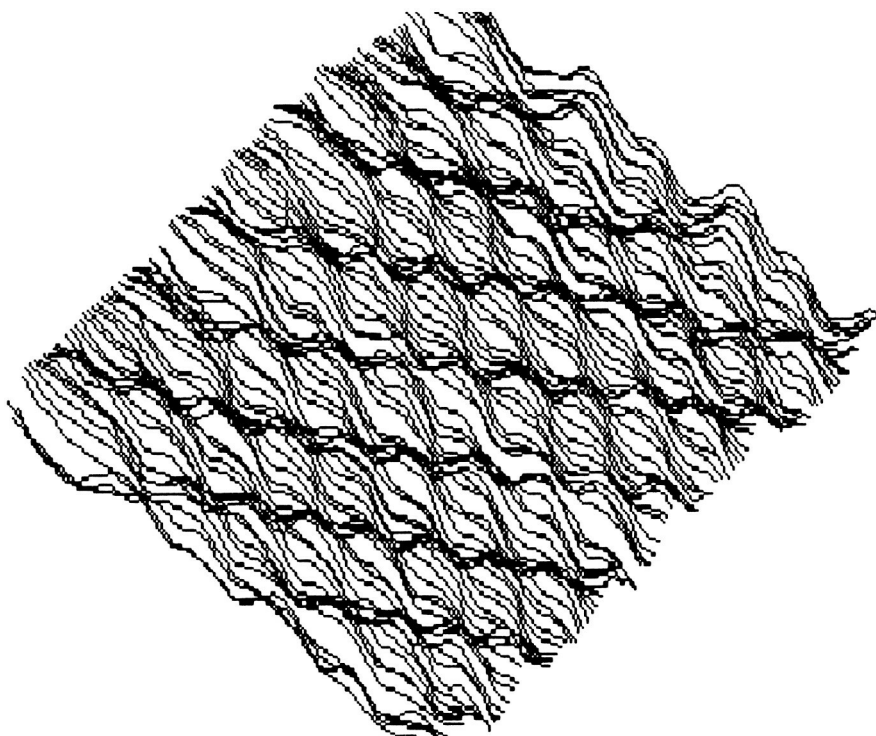
SVEC's STM addresses this problem in two ways: size and geometry. Our microscope's scanning piezo is a small cylinder of lead zirconate titanate. Voltages applied to opposing quadrants of nickel plating bend the cylinder, producing motion perpendicular to the axis, parallel to the plane of the surface. Voltages applied to all four quadrants result in motion parallel to the axis, controlling tip-to-surface separation. Earlier versions of STM had used larger piezo tripods to generate movement in three dimensions. Another important consideration, with an eye toward *in situ* UHV surface analysis, is sample manipulation. In practice, tunneling requires sample-to-tip separation on the order of 10 angstroms. An added complication is that tip-to-sample contact damages both the tip and the sample. Historically, the sample was moved into scanning range by a second "walking" piezo tripod. Such walkers faced the same problems as the tripod scanner. They were vibration sensitive, being of comparable size, and introduced a doubling of electronic complexity. The biggest problem facing SVEC

was the lack of adaptability of the walker concept to our goal of a compact and simple method of transport while under vacuum.

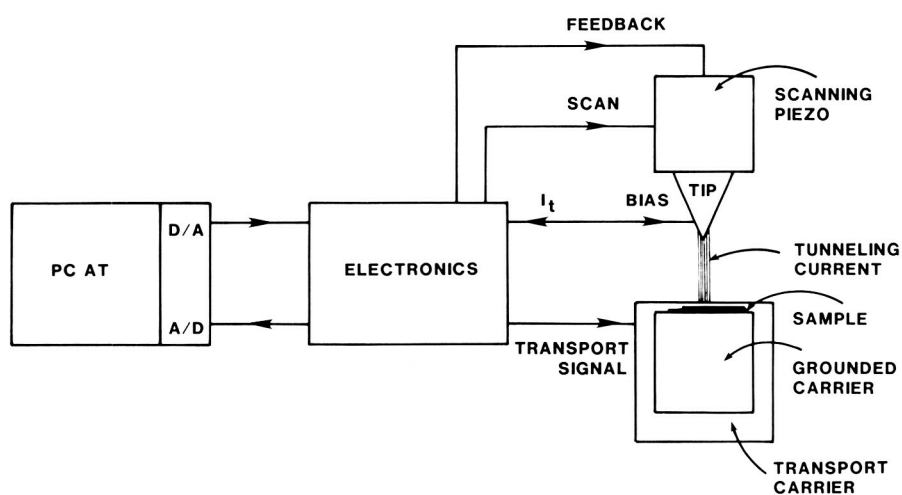
The apparent solution was disarmingly simple. A single plated piezo cylinder was mounted concentrically around the inner, scanning piezo. The outer tube has a single mode of motion along its axis (simplifying both electronics and programming). Samples are mounted on individual carriers. The carriers slide on runners mounted to the inside of the outer tube. By programming rapidly varying rates of expansion and contraction of the outer tube, the sample can be moved toward and away from the tip. The entire assembly is about one third the size of a small cigar. This combination of size and shape pushes the lowest eigenfrequency of the device well above those vibrations present even in a busy laboratory. In fact, we have successfully obtained clear images of graphite without vibration isolation of any kind in a lab crowded with activity.

Features observed by researchers using STM are a function of both the electronic states of the surface locally *and* the tip. To understand one requires a prior knowledge of the other. An asymmetrical tip will yield a distorted view of the surface under investigation. It has become clear that the scanning tip in its electronic state and stability is the lynchpin of scanning tunneling microscopy. SVEC has undertaken a detailed study of tip structure and resultant STM images to allow for detailed understanding of surface atomic structure.

Future development includes calibration of STM performance at varying high temperatures and creation of software to compensate for thermal expansion and drift, refinement of a reliable tip preparation procedure, and interfacing the current system to real-time data storage (video tape) for subsequent image processing. The result promises to be a powerful tool of unparalleled value in the surface analysis of thin films.



Scanning Tunneling Micrograph of the Graphite (0001) Surface. The hexagonal symmetry of the pattern identifies the surface atomic unit cells.



Scanning Tunneling Microscope (STM) Schematic.

THEORIES OF HOT ELECTRON TRANSPORT

ONE RESEARCH TEAM at SVEC has been commissioned to study fundamental theories of hot electron transport, current fluctuation, and noises for ultra-small-sized semiconductor systems. These studies are intimately related to the development of high speed semiconductor devices which may be used in the next generation of supercomputers and in telecommunications.

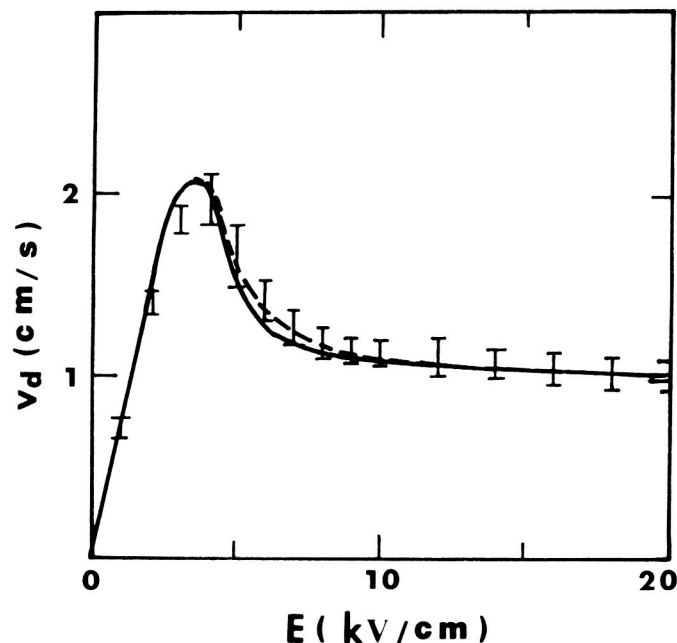
To date, investigators have focused on a number of theoretical problems of which the following five exemplify the complexity of the task ahead:

1. *Electron Transport in GaAs/AlGaAs Tunneling Junctions with Optical Phonon Emission.* Studies treat intriguing oscillatory current-voltage behavior with periods of the longitudinal optical-phonon (LO-phonon) in tunnel junctions. The origin of this oscillation is attributed to a one-dimensional localization.

2. *Balance Equation Approach to Hot Electron Transport for Many-Valley Semiconductors—Comparing with the Monte Carlo Results for n-Si.* Most of the theoretical approaches to high field electron transport in many-valley semiconductors have been based either on Monte Carlo simulation or on solving the phenomenological Boltzmann equation in relaxation time approximation. Although the analytic Boltzmann equation method has been applied to various kinds of problems in high field transport, its validity in comparison with other methods has not been established. Taking the same set of parameters as those in the Monte Carlo simulation for *n-type Si*, SVEC investigators found that their results are in excellent agreement with those of the Monte Carlo method in a wide range of temperatures and fields.

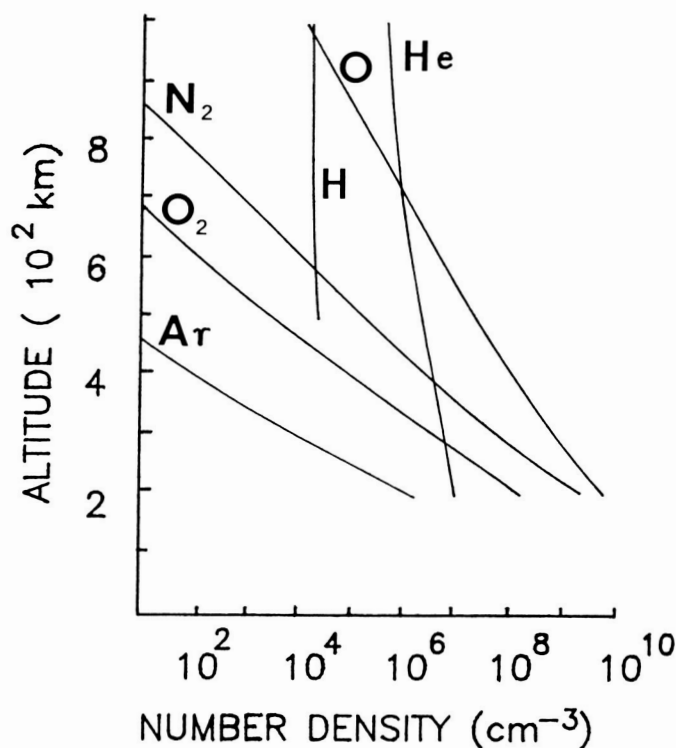
3. *Quantum Thermal Noise of Electrons in Semiconductors under Crossed Magnetic and Electric Fields.* The thermal noise of an electron system in steady-state transport under crossed magnetic and electric fields is under study using a Langevin-type equation. The formulations of noise are given for both thermal equilibrium and non-equilibrium states. Magnetic effects attributed both to the Hall effect and the magnetic quantization state have been considered. In the high-electric-field region, results show unusual noise phenomena for hot electrons.

4. *Analytic Approach to Diffusion of Hot Carriers in n-type GaAs with Γ -L-X Band Structure.* This study focuses on analytical Green's function approach to the diffusion coefficient of hot electrons in many-valley semiconductors at high electric fields. A set of Langevin-type equations is derived to calculate correlation functions for velocity fluctuation and to determine the thermal diffusion term. Investigators obtain reasonable agreement between calculated and measured diffusion coefficients when this new method is applied to *n-type GaAs* at room temperature.



Drift velocity v_d versus electric field strength E for *n-type GaAs* using Γ -L-X valleys model at $T=300\text{K}$. The bars are experimental data.

5. *Consistent Path-Integral Study for a Single Electron in a Thermal Crystal with an Applied Electric Field.* Investigators have focused on a single electron drifting in a thermal crystal under an applied electric field with the applied field and the electron-photon interaction being treated nonperturbatively. They have derived a set of coupled equations to describe with consistency the drift motion and fluctuations and have obtained various aspects of nonlinear non-equilibrium electron transport properties.



Constituent number density as a function of altitude for the terrestrial atmosphere.

IONIZED GAS AND ENERGETIC PARTICLE ENVIRONMENT FOR THE WAKE SHIELD FACILITY

ACTIVITIES OF THE PAST TWELVE MONTHS have been aimed at characterizing the ionized gas (plasma) and energetic particle environment that will be encountered by experiments placed on the WSF. Particular attention has been paid to identifying the problems that could compromise the quality of the films grown in orbit by the facility. This work has been divided into two parts: (1) study of the fluxes of suprathermal and energetic ions and neutral atoms and (2) study of the plasma wake of the orbiter.

Energetic Particle Studies

The study of energetic particles and their possible detrimental effect on the films being grown in space was based on a design objective of a film defect density an order of magnitude better than can be obtained on Earth. Upper limits for the tolerable flux of particles with energies between 100 eV and 20 keV were identified to be on the order of 10^2 - 10^3 particles $s^{-1} cm^{-2} sr^{-1} keV^{-1}$ at 1 keV. Determination of whether and how often these limits were exceeded in the vicinity of the planned WSF orbits was difficult to undertake because the penetrating *MeV* background radiation of the inner radiation belt makes the necessary measurements difficult; consequently, the results are often questionable. It is clear that the calculated flux limits appear certain to exceed themselves by four orders of magnitude during periods of major geomagnetic storms. The nature of the background flux during quieter times is not well known. There is some indication, albeit controversial, that the quiet time background may be high enough to require shielding.

In the course of the study, it became apparent that available databases have not been searched for evidence of the fluxes that are of concern to WSF experiments. These databases include data from the Defense Meteorological Satellite Program (DMSP) and the Dynamics Explorer 1 (DE-1) spacecraft.

This aspect of the work has focused on the wake shield surface. The shuttle plasma wake environment can have three possibly harmful effects on the experiments on the WSF. First, diagnostic experiments that use low energy electrons can be severely affected by charging of exposed insulators in the wake region. Second, there are some stringent limits on the amount of electric current that can be used by any electron emitting diagnostic experiment. Third, improper design of the front surface of the wake shield can trap suprathermal ions and inject them into the wake region, producing a suprathermal ion flux as high as 20% of the ram flux. This focusing, if not prevented, will limit operating pressures to 10^{-11} torr.

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Robert Keith (seated) and Frederic Connan taking an SEM micrograph.

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*Science and Research Building I, home of
the Space Vacuum Epitaxy Center.*

FINANCIAL REPORT 1987-1988

THE TOTAL SVEC budget for Fy '87 was \$2,818,274, including NASA support (\$1,376,000), University of Houston cost-sharing (\$782,274), and consortium member in-kind and cash contributions (\$660,000). NASA support provided for salaries and fringe benefits, operating expenses, research expenses, permanent equipment, and indirect costs. Total expenditures from the NASA budget were \$1,376,000. University of Houston cost sharing provided funds for salaries, fringe benefits, permanent equipment, laboratory renovation, and research expenses in thin film superconductor growth. The total UH cost sharing expenditure was \$435,093. Consortium member contributions provided funding for operating expenses beyond those reimbursed by NASA, significant discounts on permanent equipment purchases, and both direct and collaborative in-kind support. Total member expenditures were \$587,713.